

Table III. Sample of Data for Sample Pairs I and 5 from Table I

Sample Pair	Alternation Interval, Minutes	Total Running Time, Hours	Accumulated Register Counts*				Net Counting Rate				Register Counts Min.			
			Gross		Back-ground	Ratio	Net		Ratio	Start of run	End of run	A	B	B
			A	B			A	B						

Data from 32-Hour Run Showing Drop in Counting Rate

Time after Start	Counting Rate	
	A	B
1 min.	92	182
10 min.	88	177
4 hr.	83.3	165.6
16 hr.	81.5	170.7
32 hr.	81.6	163.5

* Register counts $\times 128$ = actual counts.

* Estimated from short counts at beginning and end of 2-hour run.

housing removes the voltage from the phototube when the door is opened. The scaler section of the counter was unchanged except for the mounting of the three registers on the front panel and the inclusion of a scaling factor switch. The scaling factor switch allows the selection of the factor which stores the maximum number of counts on the registers without exceeding their speed limitations. (Figure 2 shows the assembled system.)

EXPERIMENTAL

Preparation of Solutions for Test Runs. A stock solution (I) of 8.5 grams of recrystallized terphenyl in 2 liters of c. p. xylene was prepared. Each cell used for counting background contained 15 ml. of this solution. Stock solution II was obtained by dissolving 20 mg. (20 μ c.) of benzoic- α -carbon-14 acid in 1 liter of stock solution I. Stock solution III was obtained by mixing equal volumes of solution I and solution II. Thus, all three stock solutions contained the same concentration of terphenyl, but different concentrations of labeled benzoic acid. Fifteen milliliters of solution II (0.30 μ c.) were pipetted into a clean cell, and 15 ml. of solution III (0.15 μ c.) were pipetted into a second clean cell in order to obtain a pair of cells with 2 to 1 ratio of activities. After each

run the solutions were discarded and the cells and the plate glass tops were washed thoroughly with xylene followed by acetone. They were then dried. No difference within experimental error was found when the cells, holders and sample positions were interchanged.

RESULTS AND DISCUSSION

Tables I and II show the precision obtained when the ratio of the activity above background of two benzoic acid samples was determined. Table III gives a sample of data from which the ratios

Tables I and II were calculated. The shorter counting intervals gave ratios which deviated somewhat less from the calculated value and from the mean. The superiority of short intervals is a rough measure of the value of the alternating current of the instrument. The counting efficiency for carbon-14 for the results in the tables was approximately 3%.

LITERATURE CITED

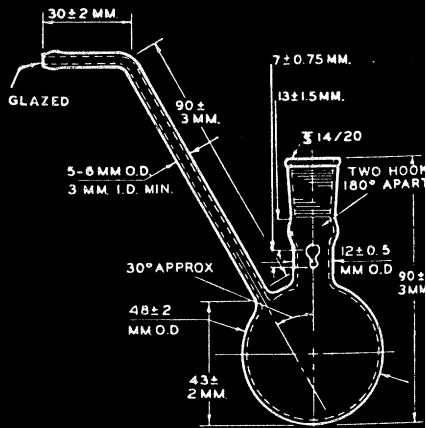
- (1) Arnold, J., *Science* 119, 155 (1954).
- (2) Farmer, E. C., and Berstein, J. A., *Ibid.*, 115, 460 (1952).
- (3) Hayes, F. N., Hiebert, R. D., and Schuch, R. L., *Ibid.*, 116, 116 (1952).
- (4) Hiebert, R. D., and Watts, R. J., *Nucleonics* 11, 38 (1953).
- (5) Neville, O. K., *J. Am. Chem. Soc.* 70, 3501 (1948).
- (6) Raaen, V. F., and Ropp, G. A., *ANAL. CHEM.* 25, 174 (1953).
- (7) Tracerlab, Inc., Boston, Mass., Tracerlog, No. 59 (April 1953).

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Report on Recommended Specifications for Microchemical Apparatus Alkoxy

IN PREVIOUS reports (1, 9, 13-17) of the Committee on Microchemical Apparatus, recommended specifications were published for pieces of apparatus which were either the most widely used for the work in question, or shown to be an improvement over the more widely used apparatus through tests made by the members of the committee and other cooperating chemists. In this report specifications are suggested for the semimicro alkoxy apparatus, which has been selected on the basis of being the most widely used.

Recommended specifications for an apparatus for the determination of alkoxy groups were delayed until a collaborative study (10, 11, 18) of methods for this determination had been conducted by the Association of Official Agricultural Chemists. In this study, compounds representing ethyl and methyl esters and ethers were submitted to practicing microchemists, who had expressed their willingness to cooperate. These individuals were asked to analyze the samples by whatever methods they were using in their own laboratories and to furnish detailed information on the procedure and apparatus. Where enough collaborators used a particular procedure or apparatus to permit the results to be treated statistically, calculations were made to determine which of these or their variations appeared to give the best accuracy and/or precision.



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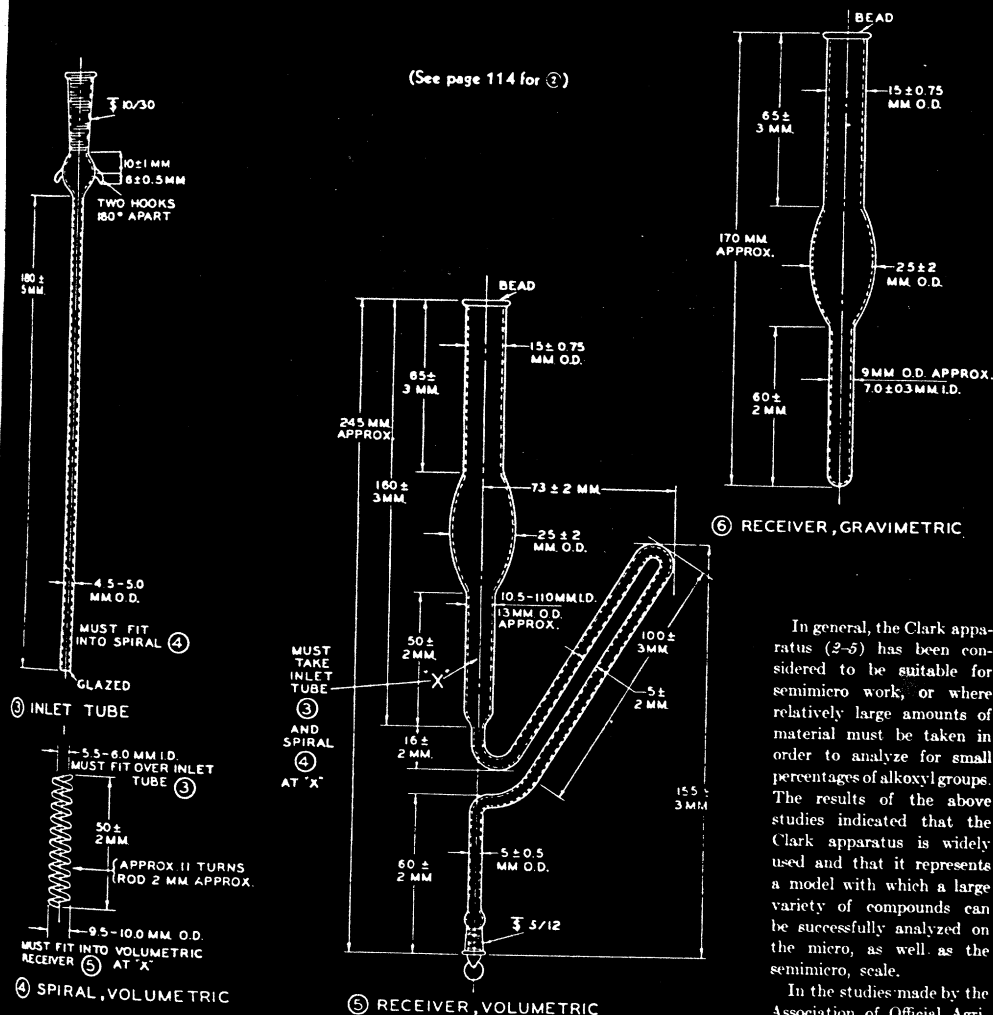
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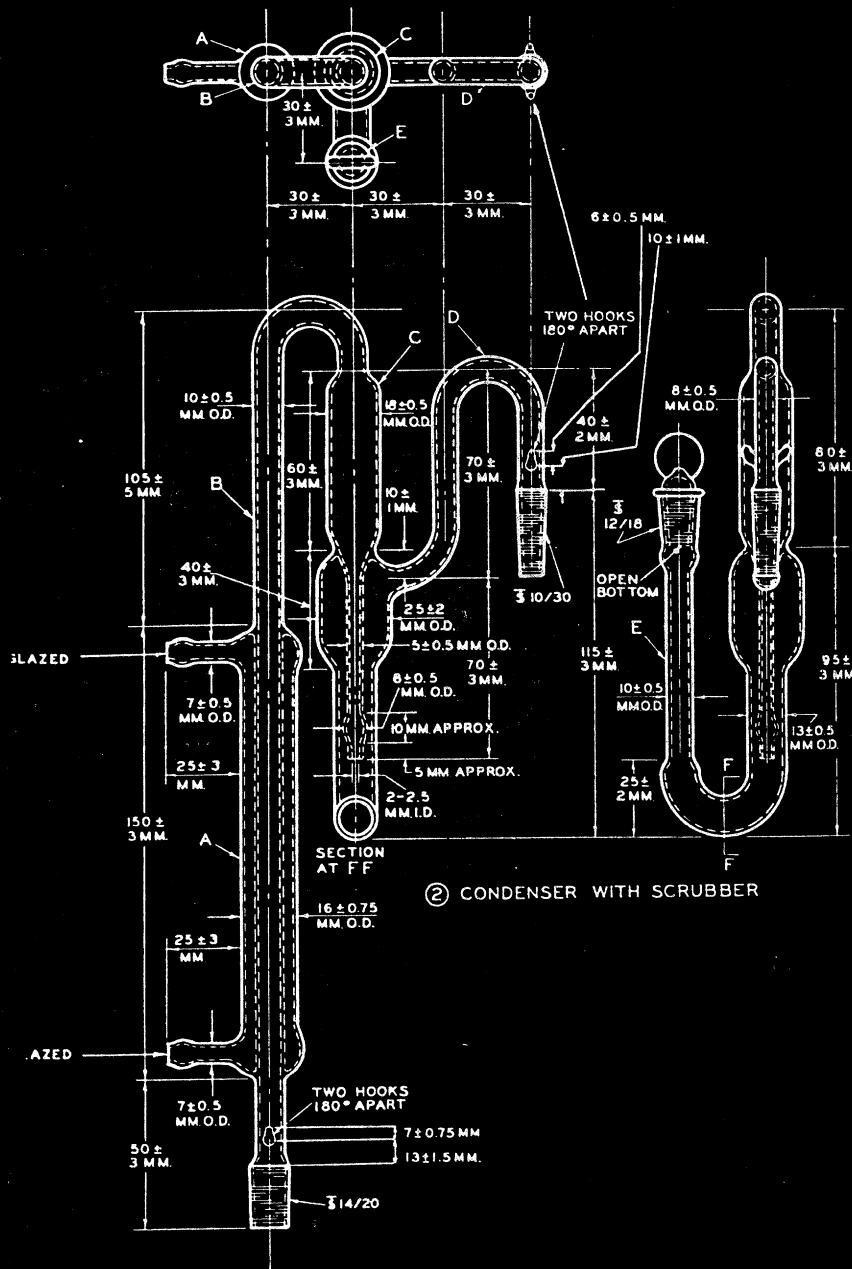
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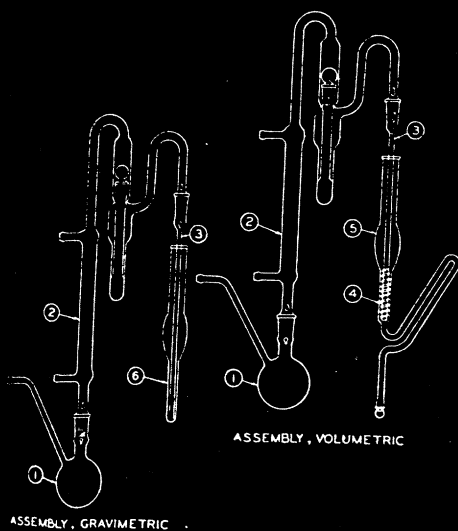
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In general, the Clark apparatus (2-5) has been considered to be suitable for semimicro work, or where relatively large amounts of material must be taken in order to analyze for small percentages of alkoxyl groups. The results of the above studies indicated that the Clark apparatus is widely used and that it represents a model with which a large variety of compounds can be successfully analyzed on the micro, as well as the semimicro, scale.

In the studies made by the Association of Official Agri-





cultural Chemists, only the volumetric procedure was used. It is well known, however, that reliable results can be obtained gravimetrically (6-8, 12). Therefore, the Committee on Microchemical Apparatus recommends specifications for a Clark-type apparatus, illustrated in the figures, which can be used for either procedure. For the volumetric procedure, the apparatus consists of the reaction flask with side arm (1), condenser with scrubber (2), inlet tube (3), spiral (4), and volumetric receiver (5) and is shown assembled. For the gravimetric procedure, the spiral (4) and volumetric receiver (5) are replaced by gravimetric receiver (6), and this is also shown assembled.

The dimensions for the side arm of the flask (1) were arrived at after a number of experiments. Capillary tubes, with and without bulbs, were unsatisfactory because of condensation in the tube. The recommended length of the side arm is necessary to minimize contact of acid with the gas connection.

The condenser with scrubber (2) has an enlarged section between the two parts to prevent suck-back of liquid from scrubber into condenser at the end of a determination. Several types of scrubbers were tested, including one constructed of two compartments connected by a capillary tube. The one selected operated more efficiently than all others tried.

The section between the scrubber and the inlet tube (3) was designed to prevent liquid being carried into the receiver. Use of the spiral (4) in the receiver (5) is optional in the volu-

metric procedure. Extensive tests have shown that equally good results are obtained without the spiral.

This apparatus was used in the collaborative study conducted by the Association of Official Agricultural Chemists this year (11), and good results were obtained by the 13-collaborating microanalysts who reported a total of 198 determinations on four samples [benzocaine (ethyl *p*-aminobenzoate), *p*-ethoxybenzoic acid, methyl *p*-aminobenzoate, and vanillin (4-hydroxy-3-methoxybenzaldehyde)].

Alkoxy apparatus of smaller dimensions than the one recommended has been described for the microdetermination of alkoxy groups (6-8, 12). The committee has considered these, but believes that further investigation is needed before any recommendations can be made.

LITERATURE CITED

- (1) *ANAL. CHEM.* 21, 651 (1949).
- (2) Association of Official Agricultural Chemists, Washington, D. C., "Official Methods of Analysis of Association of Official Agricultural Chemists," 7th ed., p. 744, 1950.
- (3) Clark, E. P., *J. Assoc. Offic. Agr. Chemists* 15, 136 (1932).
- (4) *Ibid.*, 22, 622 (1939).
- (5) Clark, E. P., "Semimicro Quantitative Organic Analysis," p. 68, Academic Press, New York, 1943.
- (6) Grant, J., "Quantitative Organic Microanalysis, Based on the Methods of Fritz Pregl," 5th English ed., p. 182, Blakiston, Philadelphia, 1951.
- (7) Roth, H., "F. Pregl Quantitative Organische Mikroanalyse," 5th ed., p. 215, Springer-Verlag, Vienna, 1947.
- (8) Steyermark, Al. *ANAL. CHEM.* 20, 368 (1948).
- (9) *Ibid.*, 22, 1228 (1950).
- (10) Steyermark, Al. *J. Assoc. Offic. Agr. Chemists* 33, 367 (1955).
- (11) *Ibid.*, in press (probable publication date, May 1956).
- (12) Steyermark, Al. "Quantitative Organic Microanalysis," p. 230, Blakiston, Philadelphia, 1951.
- (13) Steyermark, Al., Alber, H. K., Aluise, V. A., Huffman, E. W. D., Jolley, E. L., Kuck, J. A., Moran, J. J., and Willits, C. O., *ANAL. CHEM.* 23, 1689 (1951).
- (14) Steyermark, Al., Alber, H. K., Aluise, V. A., Huffman, E. W. D., Jolley, E. L., Kuck, J. A., Moran, J. J., Ogg, C. L., and Willits, C. O., *Ibid.*, 26, 1186 (1954).
- (15) Steyermark, Al., Alber, H. K., Aluise, V. A., Huffman, E. W. D., Kuck, J. A., Moran, J. J., and Willits, C. O., *Ibid.*, 21, 1283 (1949).
- (16) *Ibid.*, p. 1555.
- (17) *Ibid.*, 23, 523 (1951).
- (18) Steyermark, Al. and Loeschauer, E. E., *J. Assoc. Offic. Agr. Chemists* 37, 433-49 (1954).

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X-Ray Diffraction Patterns of Phenols—Correction

In the article on "X-Ray Diffraction Patterns of Phenols" [Hofer, L. J. E., and Peebles, W. C., *ANAL. CHEM.* 27, 1852 (1955)] on page 1856 in Table II the heading of the second column for 2,4-Dimethyl-6-isobornylphenol should be I/I_0 .

